

PATENT SPECIFICATION

(11) 1331988

1331988

NO DRAWINGS

- (21) Application No. 48316/70 (22) Filed 12 Oct. 1970
 (23) Complete Specification filed 10 May 1971
 (44) Complete Specification published 26 Sept. 1973
 (51) International Classification C08F 29/02
 (52) Index at acceptance

C3P 7A 7C12B 7C13B 7C13C 7C6A 7D1A 7D1B 7D1C
 7D1X 7D2B 7D3 7D8 7FX 7K2 7K7 7T2B
 B2E 248 269 339 368 372 388 38X 38Y 41X 41Y 421
 447 44Y 461 492 496 49Y 508 53Y

- (72) Inventor EMILE JOSEPH JEAN BAJOTT



(54) IMPROVEMENTS IN HOT MELT ADHESIVES

(71) We, ESSO RESEARCH AND ENGINEERING COMPANY, a Corporation duly organized and existing under the laws of the State of Delaware, United States of America, of Linden, New Jersey, United States of America, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—
 The present invention relates to hot melt adhesives, especially to those of which in their applications limitations are imposed by viscosity/temperature relationships. Typical examples of applications are carpet backing, bookbinding and paper coating. Specifically the invention relates to the application of hot melt compositions as backings for carpets. Such carpets may be manufactured by the traditional process or by newer techniques such as tufting and needling. The requirements for a satisfactory composition for backing tufted carpets are as follows:

Fibre Bonding:
 The main purpose of applying hot melt coatings is to bond the fibres in such a manner that under normal use no loops can be pulled out of the carpet.

Flexibility:
 A good flexibility is required in order to have a carpet which adapts well to the floor surface, and does not crack when bent through 180°C even at 0°C. Such a cracking may lead to moisture penetration, which should be avoided.

For or Dimensional Stability:
 The backing should be hard enough so that no deep imprint occurs when under permanent load from furniture.

Atactic polypropylene (APP), which is a by-product from the manufacture of isotactic polypropylene by the Ziegler-Natta process,

has been proposed as a constituent of hot melt compositions for carpet backing.

It can be used as the sole polymer to provide good bonding. However, APP has poor dimensional stability, therefore it cannot be used without additives. The introduction of high amounts of fillers (up to 60 wt %) improves the dimensional stability but ruins the flexibility of the hot melt. By adding aliphatic thermoplastic petroleum resins (ATPR) to the APP/filler blend, a drastic improvement in the flexibility is obtained, while still maintaining the same dimensional stability. This addition of ATPR also decreases the viscosity of the hot melt. The ATPR, which are advantageously used are those made by catalytic polymerization of steam-cracked petroleum hydrocarbons preferably with a boiling range from 30 to 80°C.

Such fractions consist mainly of olefins and diolefins, as well as cyclic diolefins and aromatics containing 5 to 7 carbon atoms. The cyclic olefins are dimerized by heating and substantially removed, whereafter the polymerization of the other components is carried out using Friedel-Craft catalysts. The polymerized product is steam stripped in order to obtain a resin with a softening point (Ring and Ball) that lies between 70 and 120°C. Such resins are mainly aliphatic with a paraffinic structure. These resins are compatible in all proportions with APP, and surprisingly these resins, though brittle at room temperature, do not affect the elasticity of the APP.

The use of APP in combination with ATPR is described in the specification of our co-pending patent application 25025/71 (Serial No. 1329915).

For many grades of APP, additions of ATPR do not permit a large enough reduction in viscosity to allow their use at normal application temperatures.

Hot melt backings are usually applied at temperatures within the range of 150–200°C. Higher temperatures are excluded because of difficulties of handling, higher costs and the

- effect on the carpet, particularly when made of synthetic fibres. It has now been found that any grade of APP having a high viscosity can be used in combination with ATPR, by first modifying the properties of the APP by controlled heating, preferably under inert atmosphere. By this means, APP can be modified in a reproducible manner to permit its use in hot melt formulations containing ATPR, without affecting adversely the other properties of the formulation such as flexibility and dimensional stability. By heating APP at temperatures of 200 to 300°C for a period of 5 to 40 minutes, preferably in an inert atmosphere, any grade of APP of a relatively high viscosity can be modified to a APP grade with a lower viscosity desirable for its application in the hot melt adhesive containing ATPR according to the invention.
- Atactic polypropylene may be made by conventional polymerization of propylene. It has a density of 0.885 g/cc maximum. It contains no more than 20%, preferably less than 10%, (crystalline) material insoluble in normal heptane when subjected to Soxhlet extraction for 24 hours. The viscosity average molecular weight of the material may vary depending upon the particular polymer employed but, in general, will range from 10,000 to 100,000 the preferred range being between 25,000 and 60,000 viscosity average molecular weight.

Although it is desirable, insofar as it is practical to do so, to employ polypropylene that is entirely amorphous and devoid of crystalline structure, it is not always possible to secure such purities particularly in commercial operations. However, for purposes of the present invention and in order to achieve the advantages obtained through the use of amorphous polypropylene, polymers containing a major proportion of amorphous polypropylene are used. As much as 20% isotactic or crystalline polymer may be present in the amorphous polypropylene; but preferably amorphous polypropylene containing at least 85% atactic polymer, i.e., not more than 15% crystalline material, and, where, possible, polymers containing at least 90% amorphous polymer and not more than 10% crystalline material are employed because these materials achieve the most beneficial characteristics in the hot melt adhesives according to the invention.

The invention is in particular applicable where APP, with an initial viscosity of 60,000 cps and more at 170°C is used in the modified form together with ATPR in the preparation of hot melt adhesives.

Generally the modification is carried out selecting the conditions so that the viscosity of the treated APP lies below 30,000 cps at 170°C.

The following comparison clearly demon-

strates the improvement obtained by the invention. 65

Initial viscosity of APP at 170°C:
800,000 cps.

Modification time at	temperatures of:	final viscosity of 60/40 APP/resin blend at 170°C	
0	170°C	60,000 cps	70
30 min.	220°C	3,000 cps	
20 min.	250°C	2,400 cps	
10 min.	250°C	8,000 cps	75
20 min.	280°C	2,000 cps	

The preparation of hot melt compositions for carpet backing is carried out in the following way:

The APP is heated in a mixing vessel to the desired modification temperature and kept at that temperature until the desired viscosity is reached. At this stage the ATPR is added to the hot batch. The blend is then conveniently kept at a temperature of 150—170°C and additional ingredients added, such as polyisobutylene, oil or other plasticizers. Once the blend is homogeneous, the inert fillers are added.

The amount of inert fillers can be as high as 60% by weight, and even as high as 80% by the case of temperature-treated APP together with the petroleum resin.

Low amounts of plasticizer improve the flexibility of the blend. Preferably a low molecular weight polyisobutylene (500—1300, liquid at room temperature is added in amounts of 0.5 to 5%, by weight, preferably 1 to 3%. Also maybe used 0.1—4% by weight, preferably 0.2—2% of a paraffinic, mineral softener oil, alone or together with polyisobutylene.

A hot melt backing film of about 2 to 3 mm thickness is applied on the tufted carpet, which represents a weight of 2 to 3 Kg/m².

Usually, APP is the main constituent, and 10 to 90 parts of aliphatic thermoplastic petroleum resins are used per 100 parts of APP, a preferred range being 50 to 70 parts.

The following general formulation for a carpet backing has been found to be satisfactory:

	parts by weight	
—APP	23	
—Aliphatic thermoplastic petroleum resins	17	115
—soft point 100°C (R & B)		
—Polyisobutylene		
mol. weight 600	2	
—Chalk	59.8	
—Antioxidant	0.2	120
	100.2	

	Hot melt viscosity at 170°C (cps) of backing	APP 6,000 cps	APP 800,000 cps Modified 280°C/10 min.
5	Flexibility (°C) of backing	20,000 -5	18,000 -5

(Lowest temperature at which the backing does not crack when a specimen of backed carpet is bent through 180°C).

	Hot melt viscosity at 170°C (cps) of backing	APP 2,000 cps	APP 800,000 cps Modified 280°C/20 min.
10	Flexibility (°C) of backing	3,800 +5	4,000 +5
	Hot melt viscosity at 170°C (cps) of backing	APP 800,000 cps non-modified	APP 800,000 cps Modified 250°C/20 min.
15	Flexibility (°C) of backing	500,000 20	6,200 0

WHAT WE CLAIM IS:—

1. A hot melt adhesive comprising atactic polypropylene that has been subjected to a heat treatment to lower its viscosity to below 30,000 cps at 170°C and an aliphatic thermoplastic petroleum resin with a softening point of 70—120°C (R & B).
2. A hot melt adhesive according to claim 1, wherein the atactic polypropylene has been heated to a temperature of 200—300°C for a period of 5—40 minutes.
3. A hot melt adhesive according to either of claims 1 and 2 wherein the heat treatment is carried out in an inert atmosphere.
4. A hot melt adhesive according to any one of the preceding claims wherein the atactic polypropylene contains no more than 20% crystalline polymer.
5. A hot melt adhesive according to any one of the preceding claims wherein atactic polypropylene with an initial viscosity of 60,000 cps and more at 170° is subjected to heat treatment.
6. A hot melt adhesive according to any one of the preceding claims wherein atactic polypropylene is the major component by weight.
7. A hot melt adhesive according to claim 6, which comprises 10—90 parts by weight of the thermoplastic petroleum resin per 100 parts by weight of atactic polypropylene.
8. A hot melt adhesive according to claim 7, which comprises 50—70 parts by weight of the thermoplastic hydrocarbon resin per 100 parts by weight of atactic polypropylene.
9. A hot melt adhesive according to any one of the preceding claims, which includes a minor amount of a plasticizer.
10. A hot melt adhesive according to any one of the preceding claims wherein a low molecular weight polyisobutylene, liquid at room temperature, is present in amounts of 0.5—5 wt. %.
11. A hot melt adhesive according to claim 10, wherein the amount of low molecular weight polyisobutylene is 1—3 wt. %.
12. A hot melt adhesive according to any one of claims 9 to 11 wherein 0.1—4 wt. % of a paraffinic mineral softener oil is present.
13. A hot melt adhesive according to claim 12, wherein the amount of paraffinic mineral softener oil is 0.2—2 wt. %.
14. A hot melt adhesive according to any one of the preceding claims, wherein up to 80 wt. % of inert filler is present.
15. A hot melt adhesive according to claim 14, wherein up to 60 wt. % of inert filler is present.
16. A hot melt adhesive according to claim 1 substantially as hereinbefore described.
17. A process of preparing a hot melt adhesive suitable for carpet backings, wherein atactic polypropylene is heat treated at a temperature of 200—300°C for a period of 5—40 minutes to lower its viscosity to below 30,000 cps at 170°C and is blended with an aliphatic thermoplastic petroleum resin with a softening point of 70—120°C (R & B) at a temperature of 150—170°C, whereafter additional ingredients, except the inert fillers, are added, the blend is homogenized and the inert fillers added.

K. J. VERYARD,
15 Suffolk Street,
London, S.W.1.,
Agent for the Applicants.

Printed for Her Majesty's Stationery Office, by the Courier Press, Leamington Spa, 1973.
Published by The Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from
which copies may be obtained.